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SPECIFICATION FOR SILICON TETRACHLORIDE, TECHNICAL

(First Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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January 1988

Indian Standard

SPECIFICATION FOR SILICON TETRACHLORIDE, TECHNICAL

(First Revision)

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(*Continued on page* 20)

Indian Standard

SPECIFICATION FOR SILICON TETRACHLORIDE, TECHNICAL

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 28 May 1987, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 Silicon tetrachloride is widely used in the manufacture of a variety of products, such as ethylsilicate, silanes, silicones and semi-conductor grade silicon.

0.3 The standard was first issued in 1979. Since this material is poisonous and corrosive, it is being revised to incorporate safety precautions and disposal instructions. Modifications have also been effected for the determination of relative density, distillation yield and free chlorine. Test sample and referee sample clauses has also been amended.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for silicon tetrachloride, technical.

2. REQUIREMENTS

2.1 Description — The material shall be a clear liquid, free from sediment and suspended matter, and shall consist essentially of silicon tetrachloride ($SiCl_4$).

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*Rules for rounding off numerical values (*revised*).

2.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR SILICON TETRACHLORIDE, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CLAUSE NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Relative density at 27°C/27°C	1·470 to 1·480	A-2
ii)	Distillation yield, corrected to 760 mm Hg	Not more than 2 percent by volume below 56°C and not less than 95 percent by volume between 56 and 59°C	A-3
iii)	Free chlorine (as Cl), percent by mass, *Max*	0·01	A-4
iv)	Iron (as Fe), percent by mass, *Max*	0·01	A-5

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as agreed to between the purchaser and the supplier. It is preferable that silicon tetrachloride be packed in galvanized iron containers rather than plain steel or cast iron drums.

3.2 Marking — The containers shall be marked with the name and grade (technical) of the material, net mass, manufacturer's name and recognized trade-mark, if any; and the month and year of manufacture.

3.2.1 All containers, in which the material is packed, stored or transported, shall be prominently and clearly marked with the following:

VOLATILE POISONOUS LIQUID
USE WITH ADEQUATE VENTILATION
AVOID PROLONGED BREATHING OF VAPOUR
AVOID CONTACT WITH FLAME OR HOT SURFACE

3.2.2 The cotainers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformiy to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacture or producers, may be obtained from the Bureau of Indian Standards.

4. SAFETY PRECAUTIONS AND DISPOSAL INSTRUCTIONS

4.1 Safety Precautions — Since silicon tetrachloride is decomposed by water to yield silicic acid and hydrochloric acid, it is quite corrosive to most metals, when water is present with it. In the absence of water, it has practically no action on iron, steel or the common metals and alloys, and can be stored and handled in metal equipment without danger.

Gas masks to be used while handling.

First Aid :

a) If the lungs are affected, remove from exposure and keep warm; in severe cases, get medical attention.

b) If skin is affected, drench with plenty of water and remove contaminated clothes.

4.2 Disposal Instructions — While disposing, instruct others to keep at a safe distance. Wear respirator and gloves. Spread soda ash liberally over to spillage and mop up cautiously with plenty of water. Run to waste, diluting greatly with running water.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and the conformity of the material to this standard shall be judged according to the procedure prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR SILICON TETRACHLORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemical' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.0 Outline of the Method — The relative density is determined using a suitable hydrometer.

A-2.1 Apparatus

A-2.1.1 *Hydrometer* — range between 1·400 and 1·600.

A-2.1.2 *Waterbath* — maintained at 27 ± 0·5°C.

A-2.1.3 *Measuring Jar* — 100 ml capacity.

A-2.1.4 *Thermometer* — 0-50°C.

A-2.2 Procedure — Place the material contained in a measuring cylinder in the water bath until its temperature is 27 ± 0·5°C, and read its density with the help of hydrometer. Conduct the experiment in a fuming cup board.

A-3. DETERMINATION OF DISTILLATION YIELD

A-3.0 Outline of the Method — The material is distilled in the specified apparatus under the prescribed conditions of heat input and rate of distillation. 100 ml of the material are distilled and percentages by volume of the distillate recovered at the specified temperatures are recorded.

A-3.1 Apparatus

A-3.1.1 *Distillation Flask* — of the shape and dimensions shown in Fig. 1 of IS : 2620-1963†.

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*Specification for water for general laboratory use (*second revision*).

†Specification for distillating flask.

A-3.1.2 *Thermometer* — A suitable partial immersion thermometer having a range of 48 to 100°C, graduated at each 0·2°C and with a maximum scale error of 0·2°C. The maximum overall length of the thermometer shall be 385 mm and steam diameter shall be 5·5 to 8·0 mm.

A-3.1.2.1 The thermometer shall bear a certificate of the National Physical Laboratory, New Delhi or any other organization recognized for such work.

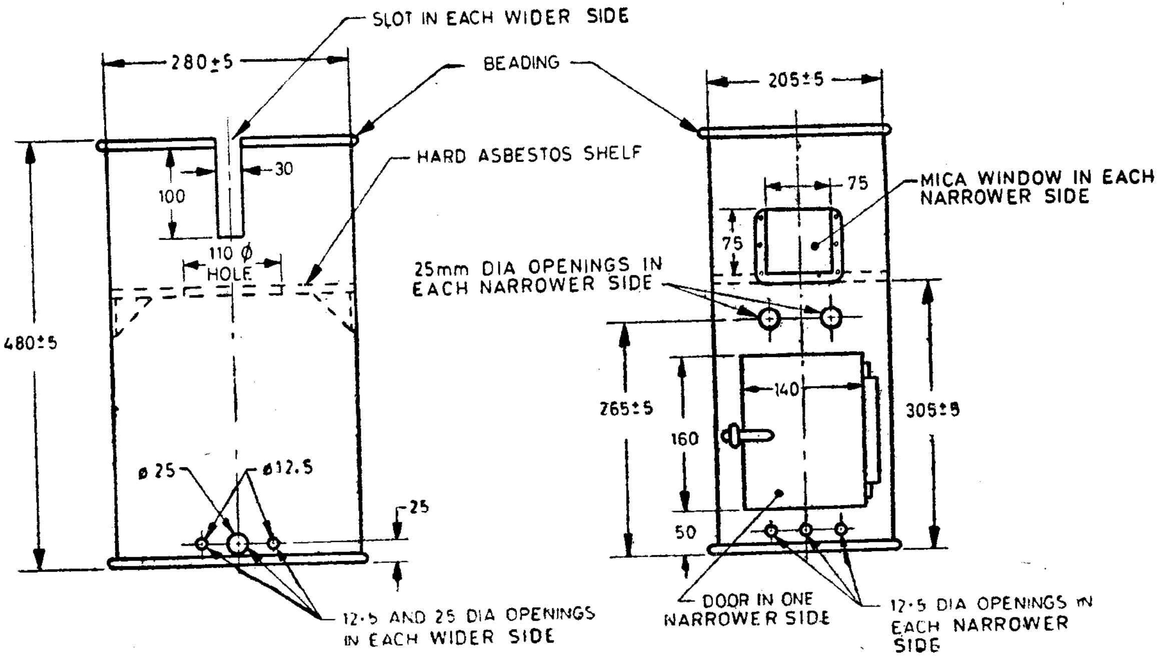
A-3.1.3 *Rectangular Draught Screen* — Rectangular in cross-section, made of 0·8 mm thick sheet metal with the dimensions shown in Fig. 1, and open at the top and bottom. It shall comply with the following requirements:

a) In each of the two narrower sides of the draught screen, there shall be two circular holes, each 25 mm in diameter and in each of the four sides of the draught screen, there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the position shown in Fig. 1. The diameter of each of the holes, centrally situated in the longer sides, shall be 25 mm and of the remaining ten holes shall be 12·5 mm. At the middle of each of the wider sides, a vertical slot with the dimensions shown in Fig. 1 shall be cut afterwards from the top of the screen. A removable shutter conforming to the dimensions shown in Fig. 2 shall be provided for closing the vertical slot not in use.

b) A shelf of hard asbestos board, 6 mm in thickness and having a central circular hole 110 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

c) In one of the narrower sides of the screen, a door shall be provided having dimensions and position as shown in Fig. 1. In each of the narrower sides of the screen, a mica window shall be placed centrally with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 1.

d) An asbestos board 150 × 150 × 6 mm in size, having a central hole 50 mm in diameter, shall be so placed on the asbestos shelf that the two holes are approximately concentric and distillation flask, when in position, completely closes the holes of the asbestos board.


SLOT IN EACH WIDER SIDE
280±5
BEADING
HARD ASBESTOS SHELF
100
30
110 ϕ HOLE
480±5
ϕ 25
ϕ 12.5
25
12·5 AND 25 DIA OPENINGS IN EACH WIDER SIDE
205±5
75
MICA WINDOW IN EACH NARROWER SIDE
75
25mm DIA OPENINGS IN EACH NARROWER SIDE
265±5
140
160
305±5
50
DOOR IN ONE NARROWER SIDE
12·5 DIA OPENINGS IN EACH NARROWER SIDE

All dimensions in millimetres.

FIG. 1 RECTANGULAR DRAUGHT SCREEN

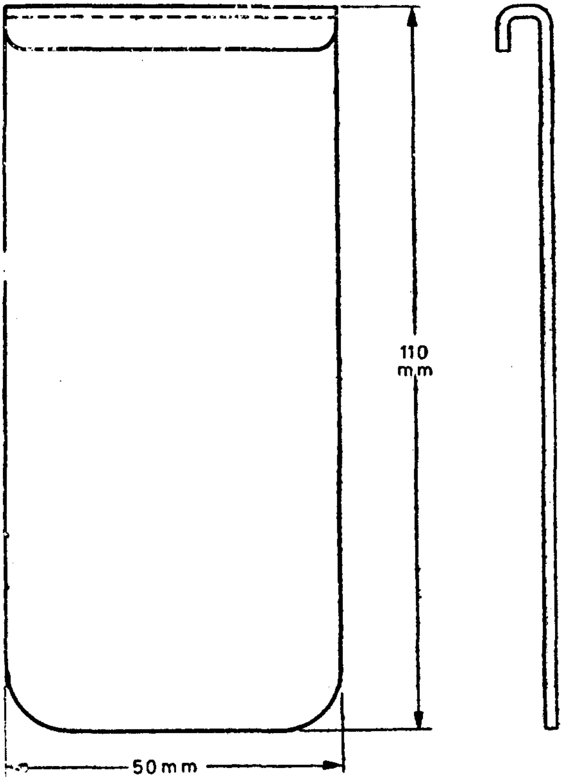


FIG. 2 REMOVABLE SHUTTER

A-3.1.4 *Liebig Condenser* — with the bent end made of Type 1 glass (graded according to IS : 2303-1963*), with a wall thickness of 1·0 to 1·5 mm and conforming to the shape and dimensions shown in Fig. 3. The bent portion may, however, be substituted by an adapter fitted externally in such a manner that distillate does not come in contact with the cork.

A-3.1.5 *Receiver* — of 100 ml capacity, with dimensions and graduations as shown in Fig. 4.

A-3.1.6 *Electric Heater, Gas Burner or Other Flame Type Heater* — Any suitable heater or burner that enables the distillation to be carried out as described in **A-3.2**.

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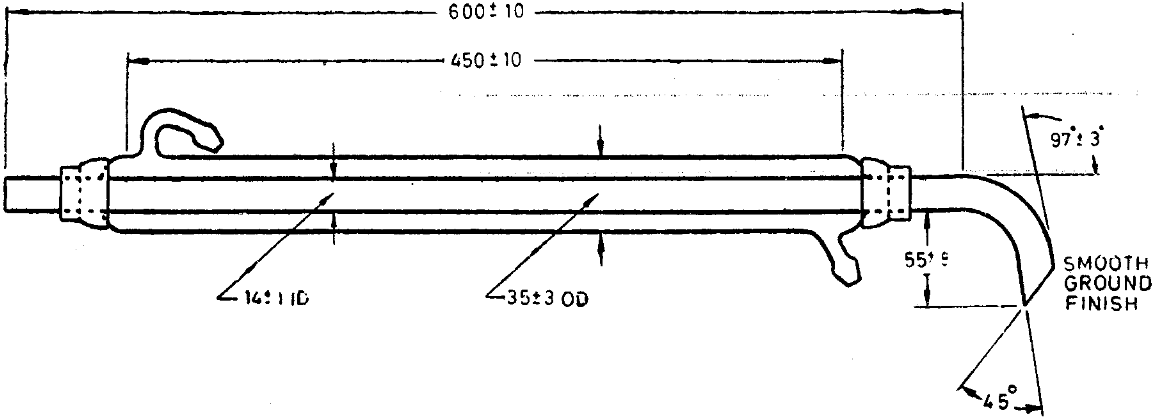
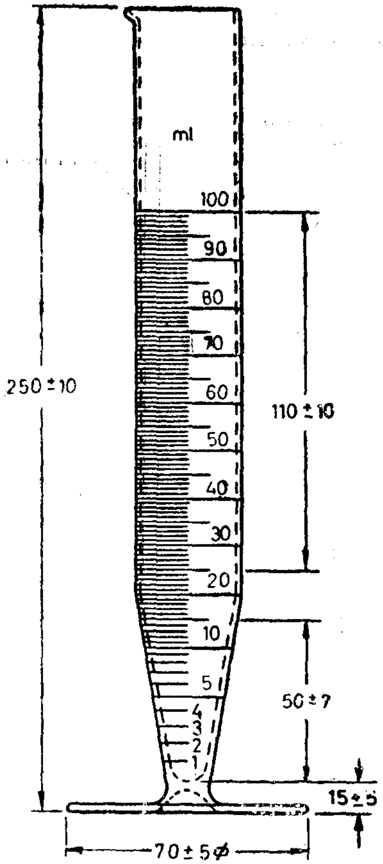
*Methods of grading glass for alkalinity.



All dimensions in millimetres.

FIG. 3 LIEBIG CONDENSER



All dimensions in millimetres.

FIG. 4 RECEIVER

A-3.2 Procedure — Assemble the apparatus as shown in Fig. 5. Measure 100 ml of the material at laboratory temperature by means of the receiver and transfer it to the distillation flask. Add a fragment (about 2 mm cube) of porous or other suitable inert material to prevent bumping, connect the flask to the condenser and insert the thermometer. Fit the thermometer in the flask so that the bottom of the capillary is in level with the lower edge of the side tube joint. Pass an adequate supply of cooling water at 0 to 5°C through the condenser. To receive the distillate, use the receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly, especially after ebullition has begun, in order that

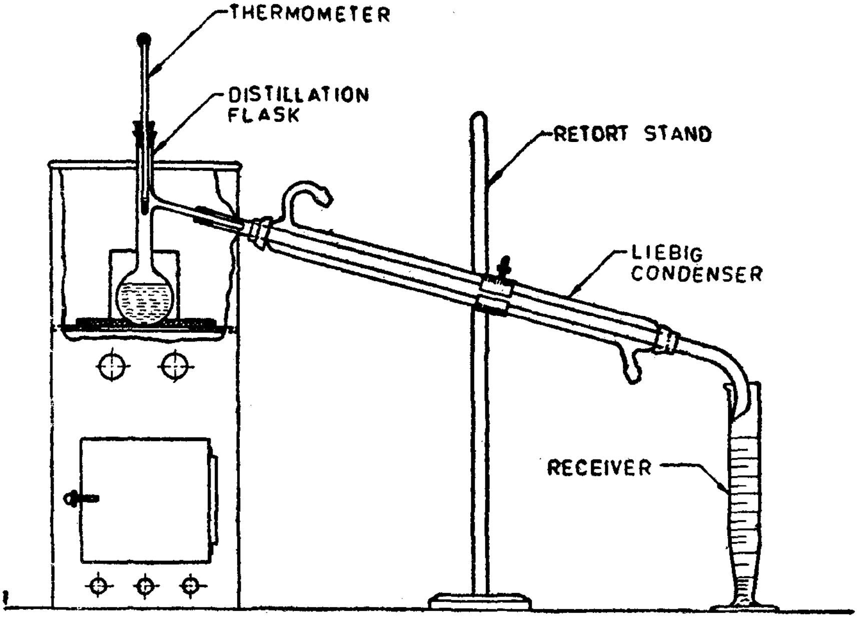


FIG. 5 ASSEMBLY OF APPARATUS FOR DETERMINING DISITILLATION YIELD

the mercury column of the thermometer may become fully expanded before the first drop of the distillate falls into the receiver, care being taken that the total period of this preliminary heating shall be not less than 5 nor greater than 10 minutes. Place the receiver in a manner that the condensate flows down its side. Continue the distillation at the rate of 4 to 5 ml per minute (about 2 drops per second). Read the volume of the distillate in the receiver, when the thermometer indicates each of the specified distillation temperatures, the temperatures on the thermometer scale being corrected as specified under A-3.3. The difference between the volumes so recorded is the percentage by volume distilling between the specified temperatures at 760 mm Hg pressure.

A-3.3 Correction of the Thermometer Reading

A-3.3.1 *Error of Scale* — In all the thermometer reading, make the corrections as indicated on the certificate of the instrument.

A-3.3.2 *Correction for Barometric Pressure* — If the barometric pressure prevailing during the determination is normal, namely, 760 mm Hg, no correction need be applied to the specified temperatures and the thermometer scale, as corrected under, **A-3.3.1** shall be used as such. If, however, the prevailing pressure deviates from 760 mm Hg, the specified temperatures shall be corrected as follows:

a) For every 10 mm Hg above 760 mm Hg, add 0·43° to the specified temperature; and

b) For every 10 mm Hg below 760 mm Hg, subtract 0·43° from the specified temperature.

A-3.3.3 For all joints in the distillation set up, apply fluoro-carbon grease. Conduct the experiment in a fuming cup board.

NOTE — These corrections are valid only for pressures above 700 mm Hg.

A-4. DETERMINATION OF FREE CHLORINE

A-4.0 Outline of the Method — A known quantity of the material is shaken carefully with potassium iodide solution and the liberated iodine is titrated against sodium thiosulphate.

A-4.1 Reagents

A-4.1.1 *Sodium Thiosulphate Solution* — 0·01 N freshly prepared.

A-4.1.2 *Starch Solution* — Triturate 5 g of pure starch and 0·01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste to one litre of boiling water; boil for 5 minutes, allow the solution to cool and decant off the clear liquid.

A-4.1.3 *Potassium Iodide Solution* — 5 percent (m/v).

A-4.2 Procedure

A-4.2.1 Take about 500 ml of water containing 10 ml of potassium iodide solution and rundown continuously 10 ml of silicon tetrachloride from a dropping pipette keeping the solution stirring vigorously with the help of a magnetic stirrer. Titrate the liberated iodine with standard sodium thiosulphate solution using starch solution as the indicator. Conduct the experiment in a fuming cup board.

A-4.3 Calculation

$$\text{Free chlorine, percent by mass} = \frac{V \times N \times 3{\cdot}55}{M}$$

where

V = volume in ml of standard thiosulphate consumed,

N = normality of thiosulphate solution used, and

M = mass in g of the material taken for test [volume multiplied by relative density (*see* **A-2**)].

A-5. DETERMINATION OF IRON

A-5.0 *Outline of the Method* — A known quantity of the material is evaporated and the residue is extracted with hydrochloric acid and estimated for iron, after removing silica.

A-5.1 Apparatus

A-5.1.1 *Platinum Crucible*

A-5.1.2 *Nessler Cylinder* — 100 ml capacity.

A-5.2 Reagents

A-5.2.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-5.2.2 *Ammonium Persulphate*

A-5.2.3 *Potassium Thiocyanate Solution* — 5 percent.

A-5.2.4 *Hydrofluoric Acid*

A-5.2.5 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in 100 ml water and 10 ml of concentrated sulphuric acid, and dilute with water to 1 000-ml mark. Transfer 100 ml of this solution to 1-litre volumetric flask and dilute to 1 000-ml mark. One millilitre of this solution is equivalent to 0·01 mg of iron (as Fe).

A-5.3 Procedure

A-5.3.1 Take 20 ml of silicon tetrachloride in a platinum dish and evaporate in a fuming cup-board. Add 5 ml of hydrofluoric acid to the residue and a few drops of sulphuric acid, and again put on a sand bath in a fuming cup-board. When all the fumes have stopped, remove the dish, heat on a light flame, cool and digest the residue with concentrated hydrochloric acid. Extract the residue and make up the volume to 100-ml with water. Pipette out 25 ml of the made up solution, add 30 mg of ammonium persulphate and boil to oxidize the iron. Cool and transfer to

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*Specification for hydrochloric acid (*second revision*).

a 100-ml Nessler cylinder, add 2 ml of potassium thiocyanate solution and dilute to 100-ml mark with water. In another Nessler cylinder, take the same amount of acid, ammonium persulphate and potassium thiocyanate solution as used with the material and dilute to about 85 ml. Add standard iron solution in small portions at a time so that after dilution to 100 ml, the colour obtained matches with that obtained with the material.

A-5.4 Calculation

$$\text{Iron (as Fe), percent by mass} = \frac{V_1}{250 \times D \times V_0}$$

where

V_1 = volume in ml of standard iron solution,

D = relative density according to **A-2.2**, and

V_0 = volume in ml of the sample taken for the test.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF SILICON TETRACHLORIDE, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 The sampling instrument shall be clean and dry.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking or stirring or both by suitable means or by rolling.

B-1.4 The samples shall be placed in suitable, clean, dry and air-tight metal or dark or amber glass containers on which the material has no action.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, and the month and year of manufacture of the material.

B-1.7 Samples shall be stored in the dark.

B-2. SAMPLING INSTRUMENT

B-2.1 The following sampling instrument may be used:

a) Sampling bottle or can, for taking samples from tanks or drums; and

b) Sampling tube, for taking samples from bottles or small containers.

B-2.1.1 *Sampling Bottle or Can* — consists of a weighted glass or metal container with removable stopper or top to which is attached a light chain (*see* Fig. 6). The bottle or the can is fastened to a suitable pole. For taking a sample, the bottle or the can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

B-2.1.2 *Sampling Tube* — made of metal or thick glass, 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 7). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.

B-2.1.2.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 For Tanks and Drums — Each tank or drum shall be sampled saparately.

B-3.2 For Bottles and Small Containers — Each lot (*see* **B-3.2.1**) shall be sampled separately.

B-3.2.1 *Lot* — In any consignment, all the containers drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-3.2.2 For ascertaining the conformity of the material in the lot to the requirements of the specification, samples shall be tested from each lot separately.

B-3.2.3 The number of containers (n) to be selected from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

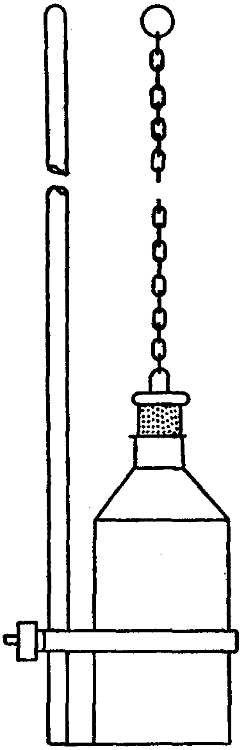
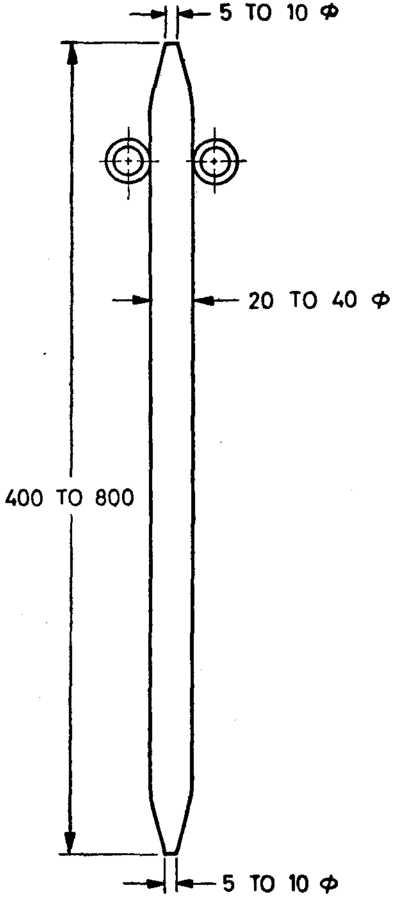


FIG. 6 SAMPLING BOTTLE OR CAN



All dimensions in millimetres.

FIG. 7 SAMPLING TUBE

B-3.2.4 The containers shall be selected at random from the lot. In order to ensure randomness of selection, reference may be made to IS : 4905-1968*. In case this standard is not available, the following procedure is recommended for use:

Starting from any container in the lot, count them in one order as 1, 2 ...... up to *r* and so on, where *r* is the integral part of *N*/*n*. Every

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*Methods for random sampling.

TABLE 2 SCALE OF SAMPLING

(*Clause* B-3.2.3)

LOT SIZE *N* (1)	NO. OF CONTAINERS TO BE SELECTED *n* (2)
Up to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

*r*th container thus counted shall be withdrawn until the requisite number of containers is obtained.

B-4. TEST SAMPLE AND REFEREE SAMPLE

B-4.1 From Tanks and Drums — As far as possible, samples from a tank or drum should be drawn during the operation of filling. In that case, equal amounts of the material shall be collected at regular intervals so as to get a total amount of about 1 000 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different depths with the sampling bottle or can, after thoroughly agitating the material so as to ensure a fair amount of homogeneity. The total amount of the material collected shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

B-4.2 From Bottles and Small Containers — From each of the bottles or containers selected according to **B-3.2.4**, a small representative portion of material shall be drawn with the help of the sampling tube. Equal quantities of the material so drawn from the various containers shall be thoroughly mixed to form a test sample of about 1 000 ml. This shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-4.3 All the test samples shall be transferred to separate sample containers, and sealed and labelled with full identification particulars. The referee test sample bearing the seals of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute. The sample bottles shall be securely packed in a box containing sufficient amount of soda ash.

B-5. NUMBER OF TESTS

B-5.1 Tests for the determination of all the requirements given in this specification shall be performed on the composite sample obtained in **B-4.1** or **B-4.2**.

B-6. CRITERIA FOR CONFORMITY

B-6.1 The lot shall be declared as conforming to this specification if all the test results satisfy the prescribed requirements.

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Chlorine and Its Inorganic Products Subcommittee, CDC 56 : 3

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